PRODUCTION OF SINTERED BODY

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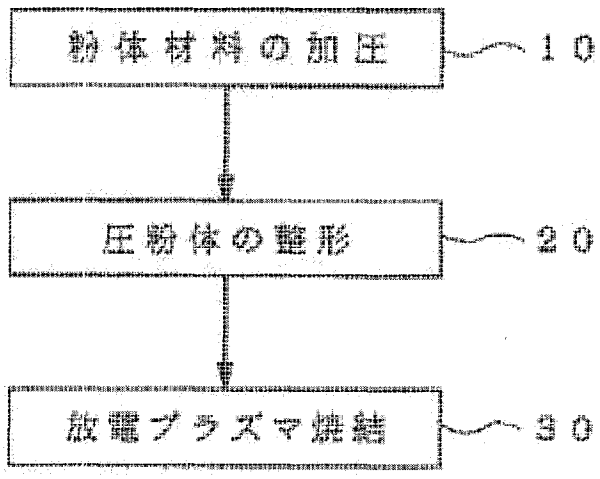
Abstract of **JP 2000128648 (A)**

PROBLEM TO BE SOLVED: To provide a method for producing a sintered body, enabling the production of the homogeneous sintered article hardly having sintering defects on the discharge plasma sintering. SOLUTION: This method for producing a sintered body comprises preparing a powder material such as ceramic powder as a raw material, isotropically adding a pressure to the powder material by a method such as a hydrostatic pressurization method (CIP, HIP) to pressure-compact the powder material, applying a mechanical processing such as a cutting processing, a grinding processing or a polishing processing to the pressurized powder article to arrange the shape or dimension, and subsequently sintering the processed powder article by a discharge plasma sintering method to obtain the sintered body.; The discharge plasma sintering is carried out by filling the pressurized powder in a molding die, pressurizing the filled powder with a pair of molding punches and simultaneously applying electric pulses to the filled material. Therein, it is preferable that a heat-insulating material is placed between the pressurized powder material and both the molding punches.



CLAIM + DETAILED DESCRIPTION

[Claim(s)]

[Claim 1] The production method of the sintered compact characterized by having the process which pressurizes granular material material isotropic and manufactures a green compact, and the process which sinters said green compact by an electric discharge plasma sintering process, and obtains a sintered compact.

[Claim 2] The production method of the sintered compact characterized by having the process which pressurizes granular material material isotropic and manufactures a green compact, the process which prepares the form or the size of said green compact, and the process which sinters the green compact operated orthopedically by an electric discharge plasma sintering process, and obtains a sintered compact.

[Claim 3] The pressurization of said granular material material is the production method of the sintered compact according to claim 1 or 2 performed by hydrostatic pressure pressurization.

[Claim 4] Said hydrostatic pressure pressurization is the production method of the sintered compact according to claim 3 performed at the temperature normal temperature or near normal temperature.

[Claim 5] The pressure of said pressurization is the production method of the sintered compact according to claim 1 to 4 which is 0.1-20tf/cm2.

[Claim 6] Said sintering is the production method of the sintered compact according to claim 1 to 5 performed by carrying out pulse energization, storing a green compact in a model and pressurizing by a pair of punches.

[Claim 7] Said sintering is the production method of the sintered compact according to claim 1 to 6 performed where thermal insulation is contacted to a green compact.

[Claim 8] Said sintering is the production method of the sintered compact according to claim 6 performed in the state where thermal insulation was made to intervene between said green compact and said punch.

[Claim 9] Said thermal insulation is the production method of the sintered compact according to claim 7 or 8 which is what consists of a non-conducting substance.
[Claim 10] Said thermal insulation is the production method of the sintered compact according to claim 7 to 9 which is a granular material whose average particle diameter is 0.05-200 micrometers.
[Claim 11] Said granular material material is the production method of the sintered compact according to claim 1 to 10 which is oxide system Ceramics Sub-Division or nitriding thing system Ceramics Sub-Division.
[Claim 12] The average particle diameter of said granular material material is the production method of the sintered compact according to claim 1 to 11 which is 0.1-200 micrometers.

[Detailed Description of the Invention]
[0001]
[The technical field to which invention belongs] This invention relates to the production method of the sintered compact manufactured by the electric discharge plasma sintering process.
[0002]
[Description of the Prior Art] It is applying effectively the high energy of the high temperature plasma which an electric discharge plasma sintering process's supplies electric direct pulse-like energy to the particle gap of granular material material, and is generated by spark discharge in an instant to thermal diffusion, electrolysis diffusion, etc. It is the technology which enables sintering or sintering junction including **** and retention time in about 5 to 20 minutes. According to such an electric discharge plasma sintering process, it is a short time and sintered compacts, such as a composite material which consists of material by which sintering was made difficult until now, or different-species material, can be obtained high-definition.
[0003] As the important section is shown in drawing 4 , the conventional electric discharge plasma sintering process With forming Di 100 made from conductive carbon One pair of forming punches 200 made from conductive carbon inserted in forming Di 100 are arranged, and it is filled up with the granular material material (sintered material) 400 in forming Di 100, and after compressing by the forming punch 200, a sintered compact is formed by energizing pulse-like current.
[0004] However, when compressing the granular material material 400 by the forming punch 200, the same distribution also as the density of the sintered compact which the granular material material 400 whole could not be compressed uniformly, but it was easy to produce the portion of ** in a part for the central part of the granular material material 400, therefore was obtained will arise. In manufacturing a sintered compact (long thing) with the long length of the

press direction of the forming punch 200 especially, there is a tendency which becomes more remarkable [the variation in the density of such a sintered compact].

[0005] And the sintered compact with such uneven density tends to produce sintering defects, such as a crack (divide) and a deficit, for the internal stress.

[0006] Moreover, pulse current flows into forming Di 100 from the forming punch 200, and makes forming Di 100 become feverish in the case of electric discharge plasma sintering. Although this generation of heat contributes to keeping warm of the sintered material 400 If the path of the forming punch 200 is extremely small as compared with forming Di 100 when comparatively big current is impressed, in order to perform rapid **** Current concentrates on the forming punch 200, this is overheated, the granular material material 400 near the press side of the forming punch 200 becomes high temperature compared with the central part, and a difference of temperature arises partially. In manufacturing a sintered compact (long thing) with the long length of the press direction of the forming punch 200 especially, there is a tendency which becomes more remarkable [the variation in such temperature distribution].

[0007] And in order to carry out the densification of the portion heated by the high temperature of the sintered compact from the other place, it becomes uneven [the density of a sintered compact] and becomes easy to produce a sintering defect as a result like the above.

[0008]

[Problem to be solved by the invention] The purpose of this invention is to offer the production method of the sintered compact which can manufacture the sintered compact which is homogeneous and has sintering defects in electric discharge plasma sintering.

[0009]

[Means for solving problem] Such a purpose is attained by this invention of following the (1) - (12).

[0010] (1) The production method of the sintered compact characterized by having the process which pressurizes granular material material isotropic and manufactures a green compact, and the process which sinters said green compact by an electric discharge plasma sintering process, and obtains a sintered compact.

[0011] (2) The production method of the sintered compact characterized by having the process which pressurizes granular material material isotropic and manufactures a green compact, the process which prepares the form or the size of said green compact, and the process which sinters the green compact operated orthopedically by an electric discharge plasma sintering process, and obtains a sintered compact.

[0012] (3) The pressurization of said granular material material is the production method of a sintered compact the above (1) performed by hydrostatic pressure pressurization or given in (2).

[0013] (4) Said hydrostatic pressure pressurization is the production method of a sintered

compact given in the above (3) performed at the temperature normal temperature or near normal temperature.

[0014] (5) The pressure of said pressurization is the production method of a sintered compact the above (1) which is 0.1-20tf/cm2, or given in either of (4).

[0015] (6) Said sintering is the production method of a sintered compact the above (1) performed by carrying out pulse energization, storing a green compact in a model and pressurizing by a pair of punches, or given in either of (5).

[0016] (7) Said sintering is the production method of a sintered compact the above (1) performed where thermal insulation is contacted to a green compact, or given in either of (6).

[0017] (8) Said sintering is the production method of a sintered compact given in the above (6) performed in the state where thermal insulation was made to intervene between said green compact and said punch.

[0018] (9) Said thermal insulation is the production method of a sintered compact the above (7) which is what consists of a non-conducting substance, or given in (8).

[0019] (10) Said thermal insulation is the production method of a sintered compact the above (7) which is the granular material whose average particle diameter is 0.05-200 micrometers, or given in either of (9).

[0020] (11) Said granular material material is the production method of a sintered compact the above (1) which is oxide system Ceramics Sub-Division or nitriding thing system Ceramics Sub-Division, or given in either of (10).

[0021] (12) The average particle diameter of said granular material material is the production method of a sintered compact the above (1) which is 0.1-200 micrometers, or given in either of (11).

[0022]

[Mode for carrying out the invention] The production method of the sintered compact of this invention is hereafter explained in detail based on the suitable embodiment shown in an accompanying drawing.

[0023] The longitudinal section showing an example of the principal part of the type for electric discharge plasma sintering by which the flowchart in which drawing 1 shows the embodiment of the production method of the sintered compact of this invention, and drawing 2 are used for electric discharge plasma sintering in this invention, and drawing 3 are the schematic views showing the example of composition of the electric discharge plasma sintering equipment used for this invention.

[0024] As shown in drawing 1 , the production method of the sintered compact of this embodiment has the pressurization process 10 of granular material material, the plastic surgery process 20 of a green compact, and the electric discharge plasma sintering process 30. Each process is explained in full detail hereafter.

[0025] [1] Pressurization of granular material material (manufacture of a green compact)
First, the granular material material used as materials is prepared. Fe system alloy material which the thing of what kind of composition is sufficient as, for example, is represented by the stainless steel of Fe or SUS304, SUS316, SUS317, SUS318, SUS410, and SUS430 grade as a granular material material, Almost all other metal material, such as nonferrous-metal system material, such as Cu or Cu system alloy, aluminum or aluminum system alloy, Ti, or Ti system alloy, is mentioned. Furthermore, Al2O3, TiO2, SiO2, oxide system Ceramics Sub-Division of ZrO2 grade, Carbide system Ceramics Sub-Division, such as SiC, Si3N4, AlN, TiN, TaN, Nitriding thing system Ceramics Sub-Division, such as BN, ****** system Ceramics Sub-Division of TiB2 grade, Various ceramic system material, such as calcium phosphate system Ceramics Sub-Division, such as fluoride system Ceramics Sub-Division, such as LiF, and hydroxyapatite, Cermet system compound material, intermetallic compound system material, an organic system material, etc. can be mentioned, one sort or two sorts or more can be mixed, and these can be used, or it can also be considered as inclination material.
[0026] Although the particle diameter in particular of granular material material is not limited, it is desirable that average particle diameter is 0.1-200 micrometers, and it is more desirable that it is 5-50 micrometers. If this average particle diameter is too small, condensation will take place and it will become what has difficult handling, and if too large, production of a green compact will become difficult.
[0027] Moreover, as a form of granular material material, not only the granular material itself but a compound, the shape of a paste, the shape of slurry, a pellet type, etc. may be what kind of form.
[0028] Pressure is applied and ****-ized to such a granular material material (or thing which carried out powder-compacting fabrication of this beforehand at desired form).
[0029] In order to make density uniform as the method of pressurization at the green compact after pressurization, the method of pressurizing isotropic, for example to a forming object like hydrostatic pressure pressurization is desirable, and especially hydrostatic pressure pressurization is desirable. This hydrostatic pressure pressurization is explained hereafter.
[0030] Although there are CIP (Cold Isostatic press) pressurized at the temperature normal temperature or near normal temperature (for example, 5-65 degrees C) and a HIP (Hot Isostatic press) pressurized under heating (for example, 65 degrees C or more) as hydrostatic pressure pressurization Since heat resistance is required by neither that equipment is simple nor the film mentioned later, the former is desirable.
[0031] As the concrete method of hydrostatic pressure pressurization, the surface of granular material material is covered with a film (not shown) with liquid interception nature, a hydrostatic pressure pressurizer is loaded with this, and hydrostatic pressure pressurization is given. In CIP, as a film, resin material, such as polyvinyl chloride, polyethylene, and polypropylene, and

rubber material like crude rubber and isoprene rubber can be used, for example. This film can be formed, for example with dipping or a vacuum packing method.

[0032] Although the pressure in particular of hydrostatic pressure pressurization (isotropic pressurization) is not limited, it is desirable that it is about two 0.1 - 20 tf/cm, and it is more desirable that it is about two 2 - 6 tf/cm. If this pressure is too low, even if it may be unable to expect the effect (especially equalization of density) of sufficient pressurization and makes pressure still higher than said upper limit, improvement in an effect will not be found, and large-sized equipment will be needed, and equipment will become expensive.

[0033] Thus, the green compact after the obtained pressurization becomes high-density, and, moreover, becomes uniform [the density]. Therefore, in order to contract uniformly while contraction percentage decreases when it sinters by an electric discharge plasma sintering process, the accuracy of dimension of the sintered compact finally obtained becomes uniform [the density of a sintered compact] highly, and generating of sintering defects, such as a crack and a deficit, is controlled.

[0034] In addition, a surface film is removed by a predetermined method after pressurization.

[0035] As mentioned above, by what granular material material is pressurized beforehand and considered as the green compact before sintering, the sintering nature of electric discharge plasma sintering improves, and it becomes high-density [the density of the obtained sintered compact], and uniform. As a result, a homogeneous and quality sintered compact is obtained. Such an effect is especially effective when a sintered compact is a long thing.

[0036] [2] Prepare the form or size if needed to the green compact obtained at the plastic surgery aforementioned process [1] of the green compact. Plastic surgery of this green compact is performed by giving predetermined machining to a green compact, for example. As machining, cutting, grinding processing, polish processing, etc. are mentioned and it can carry out combining 1 of sorts of these, and two sorts or more, for example.

[0037] Although it is desirable to be pressurized so that it may become the form and the size which are stored [that it can store inside / which the green compact manufactured mentions later at said process [1] / forming Di 1] that there is no crevice as possible, even when contrary to this, it can be considered as such form and a size by plastic surgery of a green compact.

[0038] Moreover, in order to obtain the sintered compact of a long thing, a green compact can also be orthopedically operated in the form and the size according to it. For example, one big green compact can be divided into plurality, and the following sintering processes can also be presented with each of the divided green compact.

[0039] In addition, the green compact itself can be performed, even if the hardness can perform plastic surgery of the green compact by machining etc. easily and uses the tool of low hardness especially, since it is far low compared with the sintered compact after sintering, and

its processing speed of machining is also quick.

[0040] [3] The green compact which is beyond electric discharge plasma sintering, and was made and obtained is sintered by the electric discharge plasma sintering process, and let it be a sintered compact. This sintering is performed using the electric discharge plasma sintering equipment shown, for example in drawing 3 .

[0041] Electric discharge plasma sintering of this process manufactures a sintered compact by performing pulse energization, pressurizing the green compact (sintered material) 4 with which forming Di 1 was filled up by a pair of forming punches 2. In this case, it is desirable to make thermal insulation 3 intervene between a green compact 4 and both fabrication punches 2, respectively.

[0042] As shown in drawing 2 , forming Di 1 has the shape of a cartridge which has the material insertion part 11, and one pair of forming punches 2 which fit into both ends possible [sliding] are arranged.

[0043] As a component of forming Di 1 and the forming punch 2, conductive material, such as superhard metal, a hard metal, and carbon system material (black lead, glass-like carbon, etc.), is used, for example.

[0044] Each of forming Di's 1 inner skin and press sides of the forming punch 2 is covered with the carbon sheet 5. Thereby, the forming punch 2 can be slid smoothly, without biting forming Di's 1 inner circumference. Moreover, since there is a possibility that this metal may react with the carbon contained in forming Di 1 or the forming punch 2 when metal material is included in a green compact 4, it can prevent that said metal material adheres to an inner wall surface etc. by making a carbon sheet 5 intervene.

[0045] Although such forming Di's 1 material insertion part 11 is filled up with a green compact 4, it is desirable to make thermal insulation 3 intervene between a green compact 4 and both fabrication punches 2. Diffusion of the heat from the forming punch 2 overheated by concentration of current is intercepted by installation of this thermal insulation 3, and local heating of a green compact 4 and high temperature-ization are prevented by it. As a result, the temperature of a green compact 4 is equalized and a homogeneous and quality sintered compact can be obtained.

[0046] As for thermal insulation 3, it is desirable that it is a non-conducting substance. In order that current may hardly flow through the inside of a non-conducting substance, there is no possibility that near the contact surface of thermal insulation 3 and a green compact 4 may be overheated.

[0047] What has heat resistance as a component of thermal insulation 3 is desirable, is a relation with the granular material material which constitutes a green compact 4, and is chosen suitably. For example, the difficulty sintering nature substance which is not sintered at the sintering temperature of a green compact 4 can be used as a component of thermal insulation

3.

[0048] Although what various Ceramics Sub-Division, such as oxide system Ceramics Sub-Division, nitriding thing system Ceramics Sub-Division, carbide system Ceramics Sub-Division, and fluoride system Ceramics Sub-Division, a cermet, etc. were mentioned, for example, and mixed one sort or two sorts or more for these as such thermal insulation 3 can be used That which is mainly especially concerned with either among oxide system Ceramics Sub-Division and nitriding thing system Ceramics Sub-Division is more desirable.

[0049] It decomposes easily also under high temperature, or such material does not almost have gasifying or carbonizing, and does not check s ntering of a green compact 4. Moreover, in order not to forming weld [forming Di 1 or] 2, it excels also in handling nature.

[0050] As oxide system Ceramics Sub-Division, although Al2O3, TiO2, and ZrO2 grade are mentioned, for example, especially Al2O3 is desirable. The melting point of Al2O3 is thermally [it is high and] stable, and electric insulation resistance is highly excellent as a material of thermal insulation. Furthermore, since it is stable to acid and alkali, there is no possibility of having a bad influence on formation of a sintered compact.

[0051] Although BN, Si3N4, AlN, TiN, etc. are mentioned as nitriding thing system Ceramics Sub-Division, since it is the same as that of said Al2O3, BN is desirable.

[0052] Although it is not limited especially as a form of thermal insulation 3 but a granular material, block objects, those mixtures, etc. are mentioned, it is desirable that it is a granular material. It is because the charge and extraction into forming Di 1 are easy for a granular material and regulation of the amount of charge is also easy. Moreover, a granular material is demonstrated much more effectively [a heat insulation operation] by including air between granular materials.

[0053] Moreover, thermal insulation 3 may be a block object. By considering it as a block object, mixing to the green compact 4 of thermal insulation 3 etc. can be prevented effectively.

[0054] In the case of the thermal insulation 3 by a granular material, as for the average particle diameter of this granular material, it is desirable that it is 0.05-200 micrometers, and it is more desirable that it is 0.1-100 micrometers. Furthermore, as for the average particle diameter of this granular material, it is desirable that it is 50-80 micrometers at the point that the one where particle diameter is moderately larger is excellent in the adiabatic efficiency as thermal insulation. By using the granular material of such average particle diameter, adiabatic efficiency is demonstrated more effectively and handling nature is also good.

[0055] Although the amount of charge in particular into forming Di 1 of thermal insulation 3 is not limited but it can set up suitably with sintering temperature, the character of a green compact 4, etc. It is desirable to consider it as about 8 to 70% to the weight of a green compact 4 as a quantity which can acquire required and sufficient adiabatic efficiency, and it is more desirable to consider it as about 15 to 50%.

[0056] Although thermal insulation 3 may be made to intervene respectively between a green compact 4 and the up-and-down (both ends) forming punch 2 or may be made to be placed only between one side of a green compact 4 as shown in drawing 2 , its former one is more desirable. In that case, as for the thermal insulation 3 of the quantity which is equivalent to about 8 to 70% to the weight of a green compact 4, it is desirable to be mostly distributed to division into equal parts.

[0057] The carbon sheet 5 is inserted between thermal insulation 3 and a green compact 4. Interactions, such as welding with thermal insulation 3 and a green compact 4, can be prevented by this, and thermal insulation 3 can be easily taken out after sintering.

[0058] Electric discharge plasma sintering puts thermal insulation 3 and a green compact 4 into forming Di 1, sandwiches them by the forming punch 2, and is performed by setting in electric discharge plasma sintering equipment 7 as shown in drawing 3 .

[0059] Electric discharge plasma sintering equipment 7 has the vacuum chamber 76, the pressurization rum 74 and 75 of one pair of upper and lower sides, the power supply 72 for sintering that generates pulse voltage, the hydraulic pressurization drive mechanism 73 which carries out the rise-and-fall drive of the pressurization rum 74 and 75, and the control part 71 which controls these.

[0060] Forming Di 1 who put in thermal insulation 3 and a green compact 4 is set between the pressurization rum 74 and 75 in the vacuum chamber 76.

[0061] The inside of the vacuum chamber 76 is deaerated by the vacuum pump 77, and it changes it into a vacuum state (decompression state). Since electric discharge plasma sintering has a possibility of oxygen in the air, nitrogen, water, etc. reacting with the metallic powder contained in a green compact 4, and having the influence which is not desirable on a sintered compact, it is desirable to sinter beforehand by changing the inside of the vacuum chamber 76 into a vacuum state (decompression state). Or it is desirable to sinter by making the inside of the vacuum chamber 76 into inactive gas atmosphere.

[0062] The control part 71 controls the output of the power supply 72 for sintering so that the material temperature detected by the temperature sensor (thermo couple) which was installed by forming Di 1, and which is not illustrated is in agreement with the **** curve set up beforehand. Moreover, the control part 71 controls the drive of the pressurization drive mechanism 73 and the vacuum pump 77.

[0063] It is being respectively fixed to the pressurization rum 74 and 75, and the forming punch 2 of one pair of upper and lower sides is electrically connected with the power supply 72 for sintering by the electric supply terminal (not shown) prepared in the pressurization rum 74 and 75.

[0064] By the operation of the pressurization drive mechanism 73, the pressurization rum 74 and 75 is moved in the direction approached mutually, and a green compact 4 is compressed

by the forming punch 2 fixed to these.
[0065] Although the pressurization power in particular at the time of compression of a green compact 4 is not limited, about two 80 - 2000 kgf/cm is desirable, and about two 300 - 500 kgf/cm is more desirable.
[0066] To the green compact 4 compressed still with high density by compression of this green compact 4, through the forming punch 2, the seal of approval of the pulse voltage is carried out, and pulse current is energized and sintered.
[0067] The conditions in particular of the pulse voltage to impress are not limited, for example, a pulse ratio (non-energizing time: energization time) can make them 1:1 to about 12:1, and about voltage 1-10V.
[0068] Although sintering temperature is suitably set up according to composition of a green compact 4 etc., its about 300-1500 degrees C are desirable, and its about 800-1100 degrees C are more desirable. As for the retention time in this temperature, about 2 to 30 minutes is desirable, and it is more desirable. [of about 3 to 10 minutes]
[0069] As mentioned above, although the embodiment of illustration of the production method of the sintered compact of this invention was explained The cross-sectional form of the forming punch 2 which this invention is not limited to this and inserted in forming Di's 1 material insertion part 11 and forming Di 1 can be arbitrarily chosen according to the form of a sintered compact, and circular, an ellipse form, the shape of an annulus, a polygon, etc. may be what kind of form.
[0070]
[Working example] Next, the concrete work example of this invention is explained.
[0071] (Work example 1) As a granular material material, the spherical hydroxyapatite (average particle diameter: 40 micrometers) 6.0g calcinated 700 degrees C at the air furnace was prepared, by a hydrostatic pressure press (CIP), it is temperature [of 30 degrees C], and pressure 2tf/cm2, this granular material material was pressurized isotropic, and the green compact was obtained.
[0072] In addition, on the occasion of the hydrostatic pressure press, granular material material was covered with the film made from polyvinyl chloride.
[0073] Next, as shown in drawing 2 , forming Di 1 (inside diameter of 10.5mm) made from conductive carbon is filled up with the obtained green compact 4. It put respectively 1.0g of alumina (Al2O3) granular materials (average particle diameter of 0.15 micrometer) at a time (a total of 2.0g) into the both ends as thermal insulation 3, and a pair of forming punches 2 made from conductive carbon were installed so that these might be inserted.
[0074] At this time, forming Di's 1 inner skin and the press side of the forming punch 2 were covered with the carbon sheet 5, and the carbon sheet 5 was inserted also between thermal insulation 3 and a green compact 4.

[0075] Next, it sets in electric discharge plasma sintering equipment (Sumitomo Coal Mining Co., Ltd. make SPS-510L) as forming Di 1 and the forming punch 2 shown at drawing 2 . It pressurized by pressure 400 kgf/cm2 from the upper and lower sides in the vacuum, and pulse voltage (pulse conditions are 12:2) was impressed, the compression energization system was heated, and it sintered in sintering temperature [of 1050 degrees C], and retention time 10 minutes.

[0076] the form of the obtained sintered compact -- 10.1mm[in diameter] x23.0mm in length -- being rod-like (cylindrical) -- it was .

[0077] (Work example 2) The sintered compact was manufactured like the work example 1 except having set the conditions of the hydrostatic pressure press (CIP) to temperature [of 40 degrees C], and pressure 4 tf/cm2.

[0078] (Work example 3) The sintered compact was manufactured like the work example 1 except having set the conditions of the hydrostatic pressure press (CIP) to temperature [of 40 degrees C], and pressure 6 tf/cm2.

[0079] (Work example 4) As a granular material material, the spherical hydroxyapatite (average particle diameter: 10 micrometers) 6.0g calcinated 700 degrees C at the air furnace was prepared, by a hydrostatic pressure press (CIP), it is temperature [of 30 degrees C], and pressure 2tf/cm2, this granular material material was pressurized isotropic, and the green compact was obtained.

[0080] In addition, on the occasion of the hydrostatic pressure press, granular material material was covered with the film made from polyvinyl chloride.

[0081] Next, as shown in drawing 2 , forming Di 1 made from conductive carbon is filled up with the obtained green compact 4. It put respectively 1.0g of alumina (Al2O3) granular materials (average particle diameter of 60 micrometers) at a time (a total of 2.0g) into the both ends as thermal insulation 3, and a pair of forming punches 2 made from conductive carbon were installed so that these might be inserted.

[0082] At this time, forming Di's 1 inner skin and the press side of the forming punch 2 were covered with the carbon sheet 5, and the carbon sheet 5 was inserted also between thermal insulation 3 and a green compact 4.

[0083] Next, it sets in electric discharge plasma sintering equipment (Sumitomo Coal Mining Co., Ltd. make SPS-510L) as forming Di 1 and the forming punch 2 shown at drawing 2 . It pressurized by pressure 350 kgf/cm2 from the upper and lower sides in the vacuum, and pulse voltage (pulse conditions are 12:2) was impressed, the compression energization system was heated, and it sintered in sintering temperature [of 1100 degrees C], and retention time 5 minutes.

[0084] (Work example 5) The sintered compact was manufactured like the work example 4 except having set the conditions of the hydrostatic pressure press (CIP) to temperature [of 40

degrees C], and pressure 4 tf/cm2.
[0085] (Work example 6) The sintered compact was manufactured like the work example 4 except having set the conditions of the hydrostatic pressure press (CIP) to temperature [of 40 degrees C], and pressure 6 tf/cm2.
[0086] (Work example 7) The sintered compact was manufactured like the work example 3 except not using thermal insulation.
[0087] (Comparative example 1) As a granular material material, the spherical hydroxyapatite (average particle diameter: 40 micrometers) 6.0g calcinated 700 degrees C at the air furnace was prepared.
[0088] Next, as shown in drawing 4 , forming Di 100 made from conductive carbon was filled up with this granular material material 400, and a pair of forming punches 200 made from conductive carbon were installed so that this might be inserted.
[0089] Next, forming Di 100 and the forming punch 200 are set in electric discharge plasma sintering equipment (Sumitomo Coal Mining Co., Ltd. make SPS-510L). It pressurized by pressure 400 kgf/cm2 from the upper and lower sides in the vacuum, and pulse voltage (pulse conditions are 12:2) was impressed, the compression energization system was heated, and it sintered in sintering temperature [of 1050 degrees C], and retention time 10 minutes.
[0090] (Comparative example 2) As a granular material material, the spherical hydroxyapatite (average particle diameter: 10 micrometers) 6.0g calcinated 700 degrees C at the air furnace was prepared.
[0091] Next, as shown in drawing 4 , forming Di 100 made from conductive carbon was filled up with this granular material material 400, and a pair of forming punches 200 made from conductive carbon were installed so that this might be inserted.
[0092] Next, forming Di 100 and the forming punch 200 are set in electric discharge plasma sintering equipment (Sumitomo Coal Mining Co., Ltd. make SPS-510L). It pressurized by pressure 350 kgf/cm2 from the upper and lower sides in the vacuum, and pulse voltage (pulse conditions are 12:2) was impressed, the compression energization system was heated, and it sintered in sintering temperature [of 1100 degrees C], and retention time 5 minutes.
[0093] The granular material material in work examples 1-6 and comparative examples 1 and 2, thermal insulation, the pressurization conditions of granular material material, and sintering conditions are collectively shown in following Table 1 and 2.
[0094] Visual observation of the surface and the cutting plane (five places) of a sintered compact (20 pieces each) which were obtained by the <evaluation of sintering state of sintered compact> work examples 1-7 and comparative examples 1 and 2 was carried out, and the sintering state was evaluated.
[0095] This evaluation could be four steps. Namely, when transparent-ization of hydroxyapatite by a densification is uniformly accepted in a sintered compact, Or the case where transparent-

ization is not accepted at all is considered as Evaluation A (when a sintered compact is homogeneous as a whole). Partial transparent-ization was accepted conversely, it was uneven, and the case where sintering defects, such as a crack, were accepted was considered as Evaluation D, between them was divided into two steps, and the thing near Evaluation B and Evaluation D was carried out for the thing near Evaluation A to it with Evaluation C.

[0096] An evaluation result is shown in following Table 1 and 2.

[0097]

[Table 1]

表 1

	粉体材料	断熱材	静水圧プレスの条件		焼結条件			焼結状態の評価
			温度 [℃]	圧力 [tf/cm²]	温度 [℃]	圧力 [kgf/cm²]	保持時間 [分]	
実施例1	ハイドロキシアパタイト 平均粒径40μm	アルミナ粉体 平均粒径0.15μm	30	2	1050	400	10	A
実施例2	ハイドロキシアパタイト 平均粒径40μm	アルミナ粉体 平均粒径0.15μm	40	4	1050	400	10	A
実施例3	ハイドロキシアパタイト 平均粒径40μm	アルミナ粉体 平均粒径0.15μm	40	6	1050	400	10	A
実施例4	ハイドロキシアパタイト 平均粒径10μm	アルミナ粉体 平均粒径60μm	30	2	1100	350	5	A

表2へ続く

[0098]

[Table 2]

表 2

	粉体材料	断熱材	静水圧プレスの条件		焼結条件			焼結状態の評価
			温度 [℃]	圧力 [tf/cm²]	温度 [℃]	圧力 [kgf/cm²]	保持時間 [分]	
実施例5	ハイドロキシアパタイト 平均粒径10μm	アルミナ粉体 平均粒径60μm	40	4	1100	350	5	A
実施例6	ハイドロキシアパタイト 平均粒径10μm	アルミナ粉体 平均粒径60μm	40	6	1100	350	5	A
実施例7	ハイドロキシアパタイト 平均粒径40μm	なし	40	6	1050	400	10	B
比較例1	ハイドロキシアパタイト 平均粒径40μm	なし	静水圧プレスせず		1050	400	10	D
比較例2	ハイドロキシアパタイト 平均粒径10μm	なし	静水圧プレスせず		1100	350	5	D

[0099] In the work examples 1-7, a partial densification did not occur in a sintered compact, but

density was uniform, it was homogeneous and high quality without a sintering defect and the effect that the sintered compact of high accuracy of dimension was obtained were checked by each so that clearly from the result shown in Table 1 and 2. This effect of especially the work examples 1-6 especially that carried out electric discharge plasma sintering using thermal insulation is high.

[0100] On the other hand, in comparative examples 1 and 2, the portion (high-density portion) by which hydroxyapatite was made transparent was unevenly distributed near the press side of a forming punch etc., and it turned out that the density of a sintered compact is uneven. Furthermore, there were some which sintering defects, such as a crack, have generated owing to the variation in this density. Moreover, the accuracy of dimension of the sintered compact was also low compared with each work example.

[0101]

[Effect of the Invention] As stated above, according to this invention, there are few sintering defects, such as a sintered compact uniform [density] and homogeneous, especially a crack, and a quality sintered compact can be obtained. Furthermore, the accuracy of dimension of the obtained sintered compact is also high.

[0102] And since high temperature-ization more nearly local than sintering using thermal insulation is prevented, such an effect becomes more remarkable.

[0103] Moreover, such an effect is especially effective when the sintered compact to manufacture is a long thing.

[0104] Moreover, since according to this invention granular material material is pressurized and it is considered as the green compact in advance of sintering, pressure of the pressurization at the time of sintering can be made low, and the burden of electric discharge plasma sintering equipment can be eased. moreover, the thing for which the material which was [that electric discharge plasma sintering is impossible or] difficult, and the material (for example, nitriding thing system Ceramics Sub-Division, ****** system Ceramics Sub-Division, fluoride system Ceramics Sub-Division) which receives severe restrictions in the sintering conditions of electric discharge plasma sintering also manufacture a sintered compact conventionally -- it can manufacture easily especially.

[Translation done.]